7.10–7.19 (m, 10 H), 7.39–7.47 (m, 1 H), 7.56 (t, 2 H, J = 7.6 Hz), 7.71 (t, 1 H, J = 7.4 Hz), and 7.92 (d, 2 H, J = 7.6 Hz). Anal. Calcd for $C_{31}H_{27}NO_4S_2$: C, 68.74; H, 5.02; N, 2.59. Found: C, 68.81; H, 5.09; N, 2.57.

Preparation and Cycloaddition Behavior of 1,3-Bis(phenylsulfonyl)-4-isopropyl-1,3-butadiene (35). A mixture containing 0.05 mL of piperidine, 0.05 mL of glacial acetic, 2.48 g of 1,3-bis(phenylsulfonyl)-1-propene (**23**) and 0.70 mL of isobutyraldehyde in 80 mL of a 4:1 benzene–pentane mixture was heated at reflux for 40 h by use of a Dean-Stark apparatus. At the end of this time, the mixture was allowed to cool and was added to 40 mL of a saturated ammonium chloride solution. The aqueous layer was extracted with methylene chloride, and the organic extracts were washed with a saturated sodium bicarbonate solution and water and then dried over sodium sulfate. The solution was concentrated under reduced pressure, 20 mL of ether was added, and the mixture was allowed to crystallize. The resulting solid was recrystallized from methylene chloride–ether to give 2.31 g (80%) of 1,3-bis(phenylsulfonyl)-4-isopropyl-1,3-butadiene (**35**): mp 121–122 °C; IR (KBr) 3070, 2975, 1635, 1585, 1450, 1385, 1310, 1285, 1215, 1250, 1085, 975, 860, 845, 760, 725, and 695 cm^{-1}; NMR (CDCl$_3$, 300 MHz) δ 1.12 (d, 6 H, J = 6.5 Hz), 2.80–2.90 (m, 1 H), 7.19 (d, 1 H, J = 10.5 Hz), and 7.30–7.95 (m, 12 H). Anal. Calcd For $C_{19}H_{20}O_4S_2$: C, 60.63; H, 5.36. Found: C, 60.47; H, 5.02.

A solution containing 104 mg of diene **35** and 0.04 mL of *N*-benzylidenemethylamine in 7 mL of methylene chloride was heated at 25 °C for 24 h. Removal of the solvent under reduced pressure followed by silica gel chromatography of the crude residue using a 2:3 ethyl acetate–hexane mixture as the eluent gave 110 mg (80%) of 3,5-bis(phenylsulfonyl)-6-isopropyl-1-methyl-2-phenyl-1,2,5,6-tetrahydropyridine (**37**) as a white crystalline solid: mp 161–162 °C; IR (KBr) 3070, 3040, 2960, 1445, 1315, 1305, 1220, 1155, 1145, 1085, 1055, 860, 690, and 660 cm^{-1}; NMR (CDCl$_3$, 300 MHz) δ 0.89 (d, 3 H, J = 6.8 Hz), 0.97 (d, 3 H, J = 6.8 Hz), 1.97 (s, 3 H), 2.20–2.32 (m, 1 H), 3.46 (d, 1 H, J = 4.9 Hz), 4.00–4.10 (m, 1 H), 4.55 (s, 1 H), 6.30 (b s, 2 H), 6.69 (t, 2 H, J = 7.4 Hz), 6.91 (t, 1 H, J = 7.4 Hz), 7.05–7.15 (m, 3 H), 7.25–7.40 (m, 2 H), 7.42–7.70 (m, 4 H), and 7.85 (d, 2 H, J = 7.7 Hz). Anal. Calcd for $C_{27}H_{29}NO_4S_2$; C, 65.43; H, 5.90; N, 2.83. Found: C, 65.31; H, 5.92; N, 2.77.

Preparation and Cycloaddition Behavior of 1-(Phenylsulfonyl)-3-cyano-4-phenyl-1,3-butadiene (40). To a solution containing 500 mg of 4-(phenylsulfonyl)-2-butenenitrile (**39**) and 256 mg of freshly distilled benzaldehyde in 10 mL of dry benzene was added one drop of piperidine and one drop of glacial acetic acid. The reaction was heated at reflux in a Dean-Stark apparatus for 14 h. At the end of this time, the solution was taken up in ether and the organic phase was washed with water, dried over magnesium sulfate, and concentrated under reduced pressure to give a yellow solid. Recrystallization from 70% chloroform–hexane gave 620 mg (87%) yield) of pale yellow solid, mp 163–164 °C, whose structure was assigned as 1-(phenylsulfonyl)-3-cyano-4-phenyl-1,3-butadiene (**40**) on the basis of its spectral properties: IR (CHCl$_3$) 3060, 2365, 1610, 1595, 1450, 1320, 1155, 1090, 1025, 970, 845, 810, and 690 cm^{-1}; NMR (CDCl$_3$, 300 MHz) δ 6.85 (d, 1 H, J = 14.9 Hz), 7.38–7.67 (m, 8 H), and 7.85–7.96 (m, 4 H); HRMS calcd for $C_{17}H_{13}NO_2S$ 295.0667, found 295.0657. Anal. Calcd for $C_{17}H_{13}NO_2S$: C, 69.14; H, 4.44; N, 4.75. Found: C, 69.36; H, 4.51; N, 4.77.

A solution containing 250 mg of diene **40** and 100 mg of *N*-benzylidenemethylamine in 20 mL of toluene was heated at 120 °C in a sealed tube for 22 h. The solvent was removed under reduced pressure, and the crude solid that formed was recrystallized from 50% methylene chloride–ether to give 330 mg (94% yield) of a yellow solid, mp 172–173 °C, whose structure was assigned as 1-methyl-3-cyano-2,6-diphenyl-5-(phenylsulfonyl)-1,2,5,6-tetrahydropyridine (**41**) on the basis of its spectral properties: IR (CHCl$_3$) 3010, 2990, 2235, 1605, 1450, 1310, 1145, 1085, 1020, 910, 880, and 845 cm^{-1}; NMR (CDCl$_3$, 300 MHz) δ 1.85 (s, 3 H), 3.66 (m, 1 H), 4.21 (m, 1 H), 4.78 (m, 1 H), 6.52 (d, 2 H, J = 7.4 Hz), 6.99 (dd, 1 H, J = 5.1 and 1.9 Hz), and 7.10–7.95 (m, 13 H). Anal. Calcd for $C_{25}H_{22}N_2O_2S$: C, 72.44; H, 5.35; N, 6.76. Found: C, 72.27; H, 5.31; N, 6.73.

Acknowledgment. We gratefully acknowledge support of this work by the National Institute of Health (CA-26750). Use of the high-field NMR spectrometer used in these studies was made possible through equipment grants from the NIH and NSF.

Supplementary Material Available: The final positional and thermal parameters of cycloadduct 3 (5 pages). Ordering information is given on any current masthead page.

Synthesis and Properties of New π-Donor Sulfur Heterocycles

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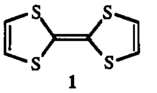
Two new π-donors related to bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF, 2) have been synthesized. These are the vinylogue 5 of BEDT-TTF and the mixed-vinylogue 4 related to TTF and BEDT-TTF.

High, one-dimensional electrical conductivity was reported for a tetracyanoquinodimethane (TCNQ) complex with tetrathiafulvalene (TTF, 1) in 1973.[1] This observation spurred a tremendous synthetic effort with the aim of discovering new and better π-donors. Subsequent work had led to scores of TTF analogues. The most investigated of these to date is bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF, 2), certain cation radical salts of which show superconducting behavior.[2-4]

One of the more interesting of the TTF-related structures is the TTF vinylogue, ethanediylidene-2,2′-bis(1,3-dithiole) (3), reported in 1983.[5] We now describe the synthesis and some properties of the mono(ethylenedithio) (4) and the bis(ethylenedithio) (5) derivatives of the TTF vinylogue 3.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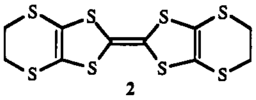
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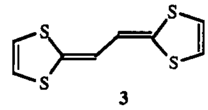
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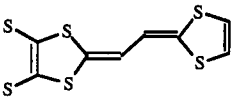
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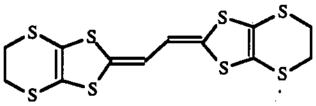
5

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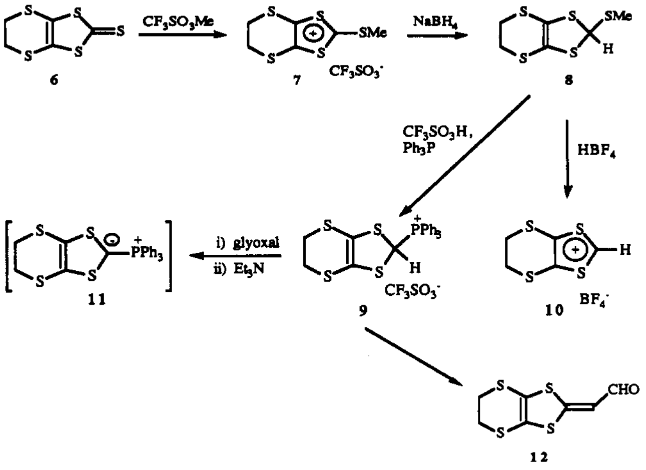
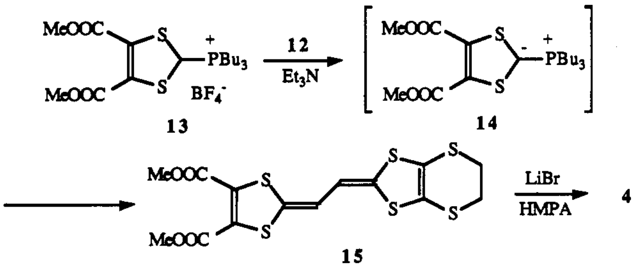
(1) (a) Ferraris, J. P.; Cowan, D. O.; Walatka, V.; Perlstein, J. H. *J. Am. Chem. Soc.* **1973**, *95*, 948. (b) Coleman, L. B.; Cohen, M. J.; Sandman, D. J.; Yamagishi, F. G.; Garito, A. F.; Heeger, A. J. *Solid State Commun.* **1973**, *12*, 1125.

0022-3263/91/1956-2720$02.50/0

Scheme I



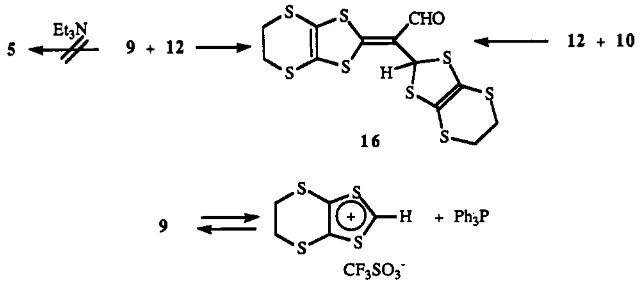
Scheme II



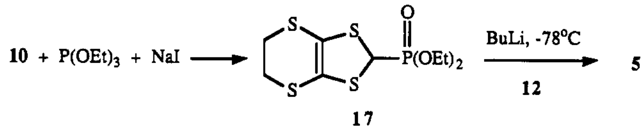
Our key starting material was the known thione 6.[2] S-Methylation of 6 was conveniently achieved by methyl triflate, which afforded the stable crystalline salt 7 in quantitative yield (Scheme I). Sodium borohydride reduction of 7 afforded the thioether 8 as a reddish oil that, without purification, was treated with triflic acid in acetonitrile, followed by triphenylphosphine, to afford the crystalline phosphonium triflate 9 in 66% overall yield. On the other hand, the intermediate thiolium ion could be isolated in 81% yield as its tetrafluoroborate 10 by reaction of 8 with HBF_4 in acetic anhydride.[6] The phosphonium salt 9 reacted with excess aqueous glyoxal in acetonitrile on addition of triethylamine to give, via the transient ylide 11, the aldehyde 12 in quantitative yield.

The known phosphonium salt diester 13[7] reacted rather sluggishly with aldehyde 12 under carefully controlled conditions to give, via ylide 14, the mixed-vinylogue diester 15 in 55% yield. Decarbomethoxylation of 15 by our LiBr–HMPA protocol[8] (Scheme II) gave the mono(ethylenedithio) vinylogue 4 in 35–40% yield.

Scheme III



Scheme IV



Attempts to carry out a Wittig-type condensation between aldehyde 12 and the phosphonium triflate 9 to give vinylogue 5 failed. The major product isolated from these reactions was a new aldehyde 16. The formation of this compound indicated that the phosphonium salt 9 was in equilibrium with a small amount of triphenylphosphine and the corresponding thiolium cation, which then added to the electron-rich α-carbon of the aldehyde. In support of this hypothesis, the thiolium salt 10 reacted with aldehyde 12 under similar conditions to give 16 in high yield. An observation similar to this one has been described earlier by us in the 1,3-diselenafulvene series[9] (Scheme III).

The synthesis of the bis(ethylenedithio) vinylogue 5 was achieved by use of a phosphonate reagent rather than a phosphonium salt. Thus, the thiolium fluoborate 10 reacted readily with triethyl phosphite in acetonitrile to give the oily phosphonate 17 in 84% yield. Reaction of 17 with n-butyllithium at −78 °C, followed by condensation with aldehyde 12, gave the desired symmetrical vinylogue 5 as sparingly soluble yellow crystals in 80% yield (Scheme IV).

The oxidation potentials of TTF vinylogues 4 and 5 were determined at room temperature in acetonitrile and 1,1,2-trichloroethane solutions, respectively. The supporting electrolyte was tetrabutylammonium hexafluorophosphate. The unsymmetrical vinylogue 4 shows two reversible oxidation waves at 0.40 and 0.57 V (SCE), compared to those shown by BEDT-TTF (2) at 0.49 and 0.74 V (SCE). As expected, the precursor 15 to the unsymmetrical vinylogue 4 showed a significantly higher oxidation potential (0.58 and 0.76 V, respectively). This clearly indicates that vinylogue 4 is a stronger electron donor than 2 and also the $E_2 - E_1$, (0.17 V) is smaller in 4 than in 2 ($E_2 - E_1 = 0.25$ V), indicative of decreased coulombic interactions in the dication derived from 4. The symmetrical vinylogue 5 also shows two reversible waves at 0.46 and 0.66 V comparable to 2.

The equilibrium geometries for the BEDT-TTF vinylogue 5 and its radical cation $5^{\bullet+}$ and for the old compound 2 and its radical cation $2^{\bullet+}$ were computed with the program package MOPAC (Version 5.04) with the PM3 parameter set[10] on the Alabama Cray X/MP-24 Supercomputer.

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The enthalpies of formation of the gas-phase species are as follows: 496.243 kJ/mol (**5**), 1214.381 kJ/mol ($\mathbf{5}^{\bullet+}$), 434.835 kJ/mol (**2**), and 1167.750 kJ/mol ($\mathbf{2}^{\bullet+}$).

The enthalpy difference (**5** − **2**) is 61.408 kJ/mol, a reasonable number for the addition of one double bond and two hydrogen atoms.

The ionization potentials of **5** and **2** are 7.984 and 7.939 eV (vertical estimates by Koopmans' theorem) and, more reliably, 7.443 and 7.596 eV (adiabatic estimates from differences in enthalpies of formation given previously). Thus, **5** is marginally easier to oxidize than **2**, in accord with the experimental result.

Presently, efforts are underway to carry out electrocrystallization of charge-transfer salts from both donors **4** and **5** in view of the unusual properties of several of the salts derived from **2**.[4]

Experimental Section

All *melting points are uncorrected.* All NMR spectra were recorded in $CDCl_3$ unless stated otherwise. The chemical shifts (δ units) are referenced against TMS as an internal standard. Elemental analyses (C, H, N, S) were carried out by Atlantic Microlabs, Norcross, GA.

Triphenylphosphonium Salt 9. A suspension of 4,5-(ethylenedithio)-1,3-dithiole-2-thione (**6**)[3] (15.0 g; 0.067 mol) in dry methylene chloride (100 mL) was treated with methyl triflate (8.5 mL; 0.068 mol; **Caution**: Methyl triflate is a powerful methylating agent. It should be handled with rubber gloves and in the hood.). The mixture was stirred under nitrogen for 4 h. A layer of dry ether (350 mL) was added. The deep reddish-orange salt **7** was filtered after 24 h, washed with more dry ether, and dried (25.7 g; ~99% yield). A solution of **7** (10 g) in MeCN (20 mL) was added to a suspension of sodium borohydride in *i*-PrOH (1.0 g in 4 mL) under nitrogen. The intense yellow color was gone. The mixture was diluted with water, and dithiole **8** separated as a heavy oil. This was extracted into ether, and the ether layer was washed with water and dried (anhydrous $MgSO_4$). Evaporation of the ether gave the crude product **8** as an oil (NMR 2.30 (s, 3 H), 2.42 (s, 6 H), 5.94 (s, 1 H)). This material was used directly in the next step. A solution of **8** in acetonitrile (80 mL) was degassed with nitrogen, cooled in an ice bath, and treated with triflic acid (4 mL) dropwise (7–8 min). To the resulting deep yellowish-orange solution was added triphenylphosphine (10.0 g). The salt **9** crystallized and was filtered after addition of dry ether (150 mL). The yield was 10.28 g (MeCN-d_3) (66.0%). Crystallization from MeCN/Et_2O gave pure **9**, mp 198 °C dec. NMR: 7.80–8.00 (m, 15 H), 6.95 (s, 1 H), 3.0 (m, 4 H). Anal. Calcd for $C_{24}H_{20}F_3O_3PS_5$: C, 47.68; H, 3.31; S, 26.49. Found: C, 47.58; H, 3.33; S, 26.42.

4,5-(Ethylenedithio)-2-(formylmethylene)-1,3-dithiole (12). Phosphonium salt **9** (10.45 g) was added to a solution of aqueous glyoxal (25 mL, 40%) in acetonitrile (175 mL). Triethylamine (4 mL) was added. The resulting yellow solution was evaporated in vacuo at 35 °C. The residue was extracted into methylene chloride and filtered quickly through a short column of silica, eluting with CH_2Cl_2–hexane (1:1). The first yellow fraction furnished the aldehyde **12**, which crystallized from methanol–water to give yellow crystals (4.0 g, 98%), mp 122 °C. MS: *m/e* 234 (M^+, 100). NMR 9.47 (d, 1 H, $J < 1$ Hz), 6.75 (d, 1 H), 3.45 (s, 4 H). Anal. Calcd for $C_7H_6OS_4$: C, 35.90; H, 2.56; S, 54.70. Found: C, 35.93; H, 2.60; S, 54.81.

Vinylogous Diester 15. To a mixture of aldehyde **12** (0.8 g) and triethylamine (1 mL) in acetonitrile (50 mL) was added (4,5-dicarbomethoxy-1,3-dithiol-2-yl)tributylphosphonium fluoborate (**13**)[7] (2.0 g) during 8 h. Diester **15** separated as deep red crystals. These were filtered (0.56 g), and by dilution of the filtrate with H_2O, a further crop of product **15** was obtained (0.14 g; total yield 55%). This was recrystallized from toluene to give red crystals, mp 142 °C. MS: *m/e* 436 (M^+, 76.3), 408 (64.1), 332 (30.7), 288 (31.7), 97 (100). NMR: 5.74 (s, 2 H), 3.82 (s, 6 H), 3.34 (s, 4 H). Anal. Calcd for $C_{14}H_{12}O_4S_6$: C, 38.53; H, 2.75; S, 44.04. Found: C, 38.58; H, 2.76; S, 44.10.

Unsymmetrical TTF Vinylogue 4. A solution of diester **15** (0.33 mg) in HMPA (6 mL) was treated with LiBr (0.32 mg) and heated at 80–90 °C for 15 min. The orange solution was heated at 150–160 °C for 15 min. An aliquot indicated the decarbomethoxylation was over. The mixture was diluted with water, and the product was extracted into benzene (3 × 50 mL). The residue (240 mg) was filtered quickly through silica in benzene solution. Crystallization of the residue from the benzene eluant gave **4** (0.14 g; 57.4%), brown crystals. An analytical sample was prepared by chromatography (Chromatotron separation) on SiO_2 with benzene–cyclohexane (1:1) to give **4** as tawny yellow crystals, mp 158 °C dec. MS: *m/e* 320 (M^+ 100) 292 (98), 150 (79.6), 172 (72), 128 (67). NMR: 6.20 (s, 2 H), 5.80 (s, 2 H), 3.35 (s, 4 H). UV: $\lambda_{max}(CS_2)$ 417 nm (log ϵ = 4.32). Anal. Calcd for $C_{10}H_8S_6$: C, 37.47; H, 2.51; S, 60.02. Found: C, 37.53; H, 2.49; S, 59.93.

Aldehyde 16. Method A. A mixture of the triphenylphosphonium salt **9** (0.25 g) and aldehyde **12** (0.17 g) in acetonitrile (30 mL) was treated with triethylamine at room temperature. After 24 h, the solvent was removed in vacuo and the residue was chromatographed (SiO_2/toluene) to give aldehyde **16** (0.08 g, 44%); crystallization from CH_2Cl_2–MeOH gave a pure sample, mp 195 °C dec. MS: *m/e* 426 (M^+, 11.2), 397 (22), 365 (55), 347 (40), 148 (58), 76 (66.2), 88 (100). NMR 9.50 (s, 1 H), 6.01 (s 1 H), 3.38 (s, 4 H), 3.31 (s, 4 H). Anal. Calcd for $C_{12}H_{10}OS_8$: C, 33.78; H, 2.36; S, 60.11. Found: C, 33.9; H, 2.36; S, 60.02.

Method B: A solution of aldehyde **12** (0.23 g) in THF (10 mL) was treated with thiolium salt **10** (0.29 g).[6] There was an instantaneous reaction. After 0.5 h, standard workup led to the isolation of aldehyde **16** (0.33 g; 77% yield), identical with the sample prepared by Method A.

BEDT–TTF Vinylogue 5. A mixture of thiolium salt **10** (1.65 g; 5.9 mmol),[6] triethyl phosphite (0.98 g; 5.9 mmol) and sodium iodide (0.89 g; 5.9 mmol) in dry acetonitrile (25 mL) was stirred under nitrogen for 0.5 h. The solvent was evaporated, and the residue was extracted into methylene chloride (75 mL). The organic extract was washed with water (2 × 50 mL) and dried, and the solvent was evaporated in vacuo. Phosphonate **17** was obtained as a heavy, slightly orange oil (1.64 g; 84%; NMR δ 4.80 (d, 1 H, J = 6 Hz), 4.22 (m, 4 H), 3.25 (m, 4 H), 1.39 (t, 6 H, J = 6 Hz)) and was used in the next step without purification. A solution of phosphonate **17** in dry THF (50 mL) was cooled to −78 °C under nitrogen in a three-necked flask fitted with a nitrogen balloon, magnetic stirrer, and a rubber septum. A solution of butyllithium (20 mL of 2.5 M in hexane) was syringed in. The color of the solution changed from red to yellow. After 5 min, a solution of aldehyde **12** (1.16 g) in dry THF (30 mL) was added slowly via the rubber septum. The reaction appeared to be instantaneous, and the product separated out. The mixture was allowed to come to room temperature, and the crystalline product was filtered, washed with ethanol, and dried (1.63 g; 80%). Recrystallization was achieved from hot DMSO when slightly greenish yellow plates were obtained, mp 245 °C dec. Alternately, **5** *could also be purified by Soxhlet extraction with* CS_2, although this appears to be time consuming. NMR: 5.98 (s, 2 H), 3.07 (s, 8 H). MS: the molecule was not volatile enough to determine EI mass spectrum; UV: $\lambda_{max}(CS_2)$ 389 nm (log ϵ, 4.52), 428 (4.56), 452 (4.57). Anal. Calcd for $C_{12}H_{10}S_8$: C, 35.09; H, 2.43; S, 62.45. Found: C, 35.15; H, 2.45; S, 62.35.

Acknowledgment. We thank the National Science Foundation (Grant No. CHE 9001714) for the support of this work. We are grateful for a gift of computer time from the Alabama Supercomputer Authority. We thank Forskerakademiet, Denmark, for a travel grant to Mr. T. K. Hansen. We also thank Mr. M. V. Joshi and Ms. R. Niziurski for the determination of CV data.

Note Added in Proof. Since the completion of our work there have been independent reports of related work: Khodorkovskii, V. Yu.; Veselova, L. N.; Neiland, O. Ya. *Khim. Geterotsikl. Soedin.* **1990**, 130; *Chem. Abstr.* **1990**, *113*, 22868. Moore, A. J.; Bryce, M. R. *Synthesis* **1991**, 27.

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